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SPECIFICATION FOR ZINC OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

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Indian Standard

SPECIFICATION FOR ZINC OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

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SPECIFICATION FOR ZINC OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 22 June 1983, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was originally published in 1964 and was subsequently revised in 1977, wherein the requirements were divided into two groups, essential and optional. Subsequently this was found non-implementable for Certification and therefore in this revision only a single set of requirements has been stipulated.

0.3 Zinc oxide issued in the manufacture of a variety of cosmetics, such as, face powders, talcum powders, depilatories, cold creams, etc.

0.4 The Sectional Committee responsible for the preparation of this standard recognized that freedom from grit is a necessary requirement for zinc oxide for cosmetic industry. In the absence of a reproducible method of test the following method may be used for testing for grit:

> Take a 20 g sample of the material in a beaker and remove by overflow under a carefully controlled steady stream of water, a larger portion of the material. The grit being heavier will remain in the beaker along with some zinc oxide. Test the pasty residue by rubbing between the finger and thumb for the presence of grit.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for zinc oxide for cosmetic industry.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a soft, white or faintly yellowish white, odourless powder. It shall be insoluble in alcohol or water and soluble in acetic acid and in mineral acids, ammonia, ammonium carbonate and alkali hydroxide solutions. It shall also be free from grit (*see* **0.4**).

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clause of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ZINC OXIDE FOR COSMETIC INDUSTRY

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. of Appendix A)
(1)	(2)	(3)	(4)
i)	Zinc oxide, percent by mass, *Min*	99·0	A-2
ii)	Arsenic (as As_2O_3), ppm, *Max*	4	A-3
iii)	Heavy metals (as Pb), ppm, *Max*	20	A-4
iv)	Sieve analysis	As agreed to between the purchaser and the supplier	A-5·1
v)	Bulk density	As agreed to between the purchaser and the supplier	A-5·2
vi)	Loss on ignition, percent by mass, *Max*	1·0	A-6
vii)	Carbonates	To pass the test	A-7
viii)	Free alkali (as NaOH), percent by mass, *Max*	0·15	A-8

---

*Rules for rounding off numerical values (*revised*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier.

3.2 Marking — The packages shall be securely closed and shall be legibly and indelibly marked with the following information:

a) Name of the material;
b) Net mass of the material;
c) Name of the manufacturer and recognized trade-mark, if any; and
d) Batch number in code or otherwise to enable the lot of manufacture to be traced back from records.

3.3 BIS Certification Marking

The product may also be marked with Standard Mark.

3.3.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative test samples of the material shall be prepared as prescribed in Appendix B.

4.2 Number of Tests

4.2.1 Tests for the determination of zinc oxide shall be conducted on each of the individual samples constituting the set of test samples (*see* **B-3.1.3**).

4.2.2 Tests for the remaining characteristics shall be conducted on the composite sample (*see* **B-3.1.2**).

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The mean and range for the test results for zinc oxide shall be calculated as follows:

Mean ($\bar{X}$) = The sum of the test results divided by the number of test results, and

Range (R) = The difference between the maximum and the minimum values of the test results.

4.3.1.1 The lot shall be declared to have satisfied the requirement for zinc oxide if the value of the expression ($\bar{X} - 0{\cdot}6\,R$) is equal to or greater than 99·0.

4.3.2 *For Composite Sample* — The test results on the composite sample shall meet the corresponding requirements specified in **2** and in Table 1.

4.3.3 The lot shall be declared as conforming to the requirements of this specification if **4.3.1** and **4.3.2** are satisfied, otherwise not.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR ZINC OXIDE FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ZINC OXIDE

A-2.0 Outline of the Method — A known mass of the material is dissolved in excess of standard acid and the excess acid is estimated by titration.

A-2.1 Reagents

A-2.1.1 *Ammonium Chloride*

A-2.1.2 *Standard Hydrochloric Acid* — 1·0 N.

A-2.1.3 *Standard Sodium Hydroxide Solution* — 1·0 N.

A-2.1.4 *Methyl Orange Indicator Solution* — Dissolve 0·01 g of methyl orange in 100 ml of water.

A-2.2 Procedure — Weigh accurately about 1·5 g of the material and 2·5 g of ammonium chloride. Dissolve in 50 ml of standard hydrochloric acid and titrate the excess of acid with standard sodium hydroxide solution using methyl orange as indicator. Carry out a blank determination without using the material.

---

*Specification for water for general laboratory use (*second revision*).

A-2.3 Calculation

$$\text{Zinc oxide, percent by mass} = \frac{4{\cdot}07\,(B - A)}{M}$$

where

B = volume in ml of standard sodium hydroxide solution used in the blank determination,

A = volume in ml of standard sodium hydroxide solution used in the titration with the material, and

M = mass in g of the material taken for the test.

A-3. TEST FOR ARSENIC

A-3.0 Outline of the Method — The stain obtained on mercuric bromide paper by arsine is compared with a stain obtained with standard arsenic solution.

A-3.1 Reagents

A-3.1.1 *Bromine Solution* — Add 30 g of potassium bromide to 30 g of bromine and add water to make the volume 100 ml.

A-3.1.2 *Brominated Hydrochloric Acid* — To 100 ml of concentrated hpdrochloric acid, add 1 ml of bromine solution.

A-3.1.3 *Stannous Chloride Solution* — Dissolve 33 g of stannous chloride in 10 ml of concentrated hydrochloric acid and add water to make the volume 100 ml. Add to this solution 100 ml more of concentrated hydrochloric acid and boil down to 100 ml. Filter before use.

A-3.2 Procedure

A-3.2.1 *Preparation of Solution* — Dissolve 1·000 g of the material in 15 ml of brominated hydrochloric acid and 45 ml of water and remove the excess of bromine with a few drops of stannous chloride solution.

A-3.2.2 Carry out the test for arsenic as directed in IS : 2088-1971* with the solution obtained in **A-3.2.1** and compare the stain obtained with that produced by 0·002 mg of arsenic trioxide.

A-4. TEST FOR HEAVY METALS

A-4.0 Outline of the Method — The colour produced by the material with hydrogen sulphide is compared with that obtained with standard lead solution.

---

*Methods for the determination of arsenic (*first revision*).

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders* — 50 ml capacity.

A-4.2 Reagents

A-4.2.1 *Concentrated Hydrochloric Acid* — *see* IS : 265-1976*.

A-4.2.2 *Ammonium Chloride*

A-4.2.3 *Acetic Acid* — 1 N.

A-4.2.4 *Standard Lead Solution* — Dissolve 1·6 g of lead nitrate in water, add 1 ml of concentrated nitric acid (*see* IS : 264-1976†) and make up the volume to 1 000 ml. Pipette out 10 ml of solution and dilute again to 1 000 ml with water. One millilitre of the solution contains 0·01 mg of lead (as Pb).

A-4.2.5 *Hydrogen Sulphide Solution* — Freshly prepared, saturated solution.

A-4.3 Procedure — Heat 0·500 g of the material in a silica crucible in a muffle furnace at 475 to 500°C. Cool and treat the residue with 3 drops of concentrated hydrochloric acid. Evaporate to dryness over a low flame and return to the muffle furnace for 20 to 30 minutes. A clean white ash should result, otherwise the hydrochloric acid treatment may be repeated. Dissolve the ash in 1 ml of dilute hydrochloric acid (1:1) and wash with several portions of water into an evaporating dish. Evaporate to dryness on steambath and dissolve the residue in about 30 ml of water. Transfer the solution to a Nessler cylinder, add 1 g of ammonium chloride and 1 ml of acetic acid. Carry out a control test in another Nessler cylinder using 1·0 ml of standard lead solution in place of the material and 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler cylinder, add 10 ml of hydrogen sulphide solution, dilute to the mark with water and shake well. Compare the intensity of the colour produced in the two cylinders.

A-4.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour obtained with material is not greater than that obtained in the control test.

A-5. DETERMINATION OF SIEVE ANALYSIS AND BULK DENSITY

A-5.1 Sieve Analysis

A-5.1.1 *Procedure* — Place about 10 g of the material, accurately weighed, in the agreed sieve and wash by means of a slow stream of

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*Specification for hydrochloric acid (*second revision*).

†Specification for nitric acid (*second revision*).

running tap water (*see* Note 1) and finally with fine stream from a wash-bottle until all the material that can pass through the sieve has passed (*see* Note 2). Let the water drain from the sieve and then dry the sieve containing the residue on a steam-bath. If there is any residue, carefully transfer it on to a tared watch-glass and dry to constant mass at 105° ± 2°C.

NOTE 1 — The tap water is usually sufficiently free from suspended particles to be utilized directly as it comes from the faucet. If the water is turbid, it is necessary to place a large bottle of distilled water above the working sink and to siphon the water through a clean rubber tube to the level of the sieve. It is a good plan to insert a small rubber stopper carrying a short glass tube about 5 cm in length and 0·6 cm in internal diameter in the faucet outlet. A thin flexible piece of clean rubber tubing (new tubing should first be thoroughly washed) about 60 cm in length is then attached to the glass tube. The faucet is turned on very slowly until a thin stream of water is coming through the rubber tube. By pressing the end of the rubber tube between the fingers, the stream of water can be varied in intensity and directed to any section of sieve. Experience has shown that the best practice is to tilt and tip the sieve from one side to another alternately, washing the sample from the higher level of the sieve to the lower.

NOTE 2 — In order to obtain comparable results, it is essential to continue washing the residue on the sieve untile one is satisfied that all the material fine enough to pass through the sieve has been removed. At least one-half hour of washing is essential in most cases. It is a good plan to catch the last washes in a beaker. By viewing the contents of the beaker against a dark coloured background, it is possible accurately to gauge the progress of the washing operations.

A-5.1.2 *Calculation*

Material retained on the specified sieve, percent by mass $= 100 \frac{m}{M}$

where

m = mass in g of the residue retained on the specified sieve, and

M = mass in g of the material taken for the test.

A-5.2 Bulk Density

A-5.2.1 *Apparatus*

A-5.2.1.1 Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder (*A*) together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error, if any, of less than one millilitre. The distance between zero and 250-ml graduation marks on the measuring cylinder (*A*) shall be not less than 220 mm and not more than 240 mm. The distance between the flat ground part of the base of measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to full height, shall be 25 ± 2 mm.

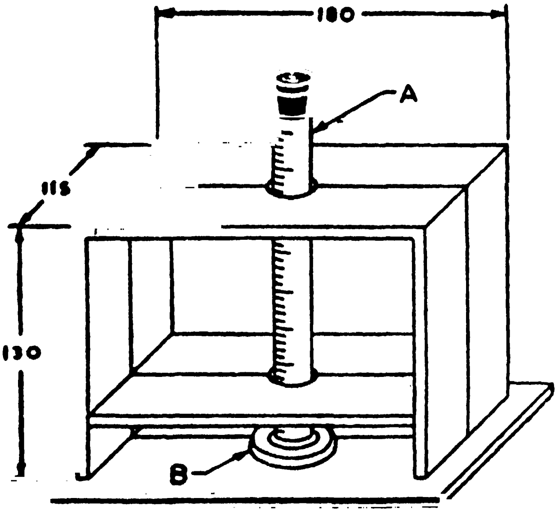

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-5.2.1.2 *Rubber base pad* — The rubber base pad *B* shall have a shore hardness of 42 to 50.

A-5.2.1.3 *Balance* — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0·1 g.

A-5.2.2 *Procedure* — Sieve about 40 g of the material through 250 micrometre IS sieve [*see* IS : 460 (Part I)-1978*] on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder and within one second lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper stop) and let it drop. Note the volume after dropping it once. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation, give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

---

*Specification for test sieves : Part 1 Wire cloth test sieves (*second revision*).

A-5.2.3 *Calculation*

Bulk density, g/ml:

a) After one tap $= \frac{m}{V_1}$

b) After 50 drops $= \frac{m}{V_{50}}$

where

m = mass in g of the material taken for the test,

V_1 = volume in ml of the material after one drop, and

V_{50} = volume in ml of the material after 50 drops.

A-6. DETERMINATION OF LOSS ON IGNITION

A-6.1 Procedure — Weigh accurately about 2 g of the material and ignite at about 600°C in a porcelain crucible, to constant mass.

A-6.2 Calculation

$$\text{Loss on ignition, percent by mass} = 100 \frac{(M_1 - M_2)}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the material after inginition.

A-7. TEST FOR CARBONATES

A-7.0 Outline of the Method — The material is treated with an acid; any effervescence is taken to indicate presence of carbonates.

A-7.1 Reagent

A-7.1.1 *Dilute Hydrochloric Acid* — approximately 0·1 N.

A-7.2 Procedure — Take about 0·5 g of the material in a 50-ml beaker and add about 10 ml of dilute hydrochloric acid. Gently warm the solution.

A-7.2.1 The material shall be taken to have passed the test if there is no effervescence.

A-8. DETERMINATION OF FREE ALKALI

A-8.0 Outline of the Method — An aqueous extract of the material is titrated with acid, using phenolphthalein as indicator.

A-8.1 Reagents

A-8.1.1 *Standard Hydrochloric Acid* — 0·1 N.

A-8.1.2 *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959* and IS : 2263-1979†).

A-8.2 Procedure — Weigh accurately about 5 g of the material and add 50 ml of hot water. Stir well the suspension for about 10 minutes. Filter by decantation and wash the residue twice with 10-ml portions of water, passing the washings again by decantation through the filter. Collect all the filtrates in a beaker. Titrate the combined filtrates with standard hydrochloric acid using phenolphthalein as indicator.

A-8.3 Calculation

$$\text{Free alkali (as NaOH), percent by mass} = \frac{0{\cdot}4\,V_1}{M_1}$$

where

V_1 = volume in ml of standard hydrochloric acid used in the titration, and

M_1 = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF ZINC OXIDE FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instruments shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

---

*Specification for rectified spirit (*revised*).

†Methods of preparation of indicator solutions (*first revision*).

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot separately for ascertaining conformity of the material to the requirements of this specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABEE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size N (1)	Number of Containers to Be Selected n (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables (*see* IS : 4905-1968*) shall be used; in case such tables are not available, the following procedure may be adopted:

---

*Methods for random sampling.

'Starting from any container, count them 1, 2, 3 .... up to *r* and so on in a systematic manner, where *r* is the integral part of N/n. Every *r*th container thus counted shall be withdrawn from the lot, till the requisite number of containers required for sampling are obtained.'

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **2** and shall be not less than 500 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 500 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third as a referee sample.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stopper and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (**B-3.1.2**) and a set of individual samples (**B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

AMENDMENT NO. 1 MARCH 2002
TO
IS 2850 : 1983 SPECIFICATION FOR ZINC OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

[*Page* 4, *Table* 1, *Sl No.* (iii)] — Substitute the following for the existing:

Sl No.	**Characteristics**	**Requirement**	**Method of Test (Ref to Cl No. of Appendix A)**
iii)	Heavy metals (as Pb)	To pass the test	A-4

(*Page* 7, *clauses* A-4 to A-4.3.1) — Substitute the following for the existing:

'A-4 TEST FOR HEAVY METALS

A-4.1 Reagents

A-4.1.1 *Acetic Acid* – 1 N.

A-4.1.2 *Potassium Chromate Solution* (5 percent, m/v).

A-4.2 Procedure – Dissolve 2 g of the material in a mixture of 20 ml water and 5 ml of glacial acetic acid. Add 0.25 ml of potassium chromate solution. The sample shall be taken to have passed the test if solution on addition of potassium chromate solution remains clear.'

(PCD 19)

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*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices::	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT BUilding, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17

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*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222 39 71

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